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378040**SYNTHESIS OF NEW FLUORINE COMPOUNDS (U)****J. B. Beal, Jr.****Final Report
December 1966****Contract AF 04(611)9556
Advanced Research Projects Agency
Washington 25, D. C.
ARPA Order Number 24**

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SYNTHESIS OF NEW FLUORINE COMPOUNDS (U)

J. B. Beal, Jr.

**Final Report
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FOREWORD

This is the final technical summary report prepared by Ozark-Mahoning Company, Tulsa, Oklahoma under contract AF 04(611)9556. This report covers only the work done in the last year 1 October 65 - 30 September 66 in as much as the work, though somewhat related, can be considered independently of that of the first two years. Furthermore, the work of the first two years was covered in a single, unclassified report dated March 1966 (AFRPL-TR-66-49) and may be obtained as report AD 480 450 by qualified users of the Defense Documentation Center.

This work has been monitored by the Air Force Rocket Propulsion Laboratory (RPCL), Edwards, California. Lt. Charles M. Stone is the Project Engineer. Capt. Joel A. Tolson and 1st Lt. Eugene Irene also served as Project Engineers.

The program was sponsored by the Advanced Research Projects Agency, Washington 25, D. C. under ARPA Order Number 24, Amendment Nr. 64, Task Nr. 15.

Dr. J. B. Beal, Jr., is the Project Scientist and is assisted by Mr. Karl Schmidt.

Thanks are due to Dr. E. F. Cain and his associates at Rocketdyne for their efforts in supplying a small sample of ClF_5 .

This report has been reviewed and is approved.

WILLIAM H. EBELKE
Colonel, USAF
Chief, Propellant Division

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ABSTRACT

Reactions were studied in which high pressure and elevated temperatures were employed. Pressures ranged from 340 to about 680 atm.

The reactions studied were as follows: OF_2 with CsClF_4 , OF_2 with ClF_3 , OF_2 with N_2F_4 , CsF with NF_3 , fluorine with NF_3 , and CsF and fluorine with NF_3 .

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I. INTRODUCTION AND SUMMARY

The objective of this program has been the synthesis of new compounds comprised of fluorine, oxygen, and nitrogen through the use of high pressure reaction conditions. The types of products envisioned were those which would be quite likely to have high potential as propellant oxidizers. The reactions of specific interest that were studied are as follows:

1. OF_2 with CsClF_4
2. OF_2 with ClF_3
3. OF_2 with N_2F_4
4. CsF with NF_3
5. F_2 with NF_3
6. CsF and F_2 with NF_3

High pressure means, in this case, the range between 5,000 and 15,000 psia. The temperature range varied between ambient room temperature and 350°. The pressures actually used were about 5,000 and about 10,000 psia although the autoclaves were rated for 15,000 at 350°.

The new products sought from the first two reactions are those in which one or more of the fluorine atoms in ClF_5 are replaced by an O-F group, e.g., F_4ClOF . Another type of product that could have formed in these reactions and would have been of considerable interest was the dioxygenyl salt, O_2ClF_4 . Our earlier work revealed that OF_2 decomposes and forms the O_2F^\cdot radical which will in turn react with certain Lewis acids to form the dioxygenyl salt (1). Finally, one can imagine that compounds such as ClF_5 , ClF_3O , and ClF_7 might be products.

The third reaction listed was studied in hopes that a hypofluorite of nitrogen might be formed.

The last three reactions were studied in an attempt to extend an analogy from chlorine to nitrogen with the resultant product being NF_5 .

The results of the reactions are summarized as follows:

1. When OF_2 is allowed to react with either CsClF_4 or ClF_3 a number of products are formed depending on the conditions and the reactor used. The reactors used were constructed of either Monel or Hastelloy-C. The effect of pressure appears to hold less significance than that of temperature. The reactions do seem to proceed in much the same way as would be expected if a

mixture of oxygen and fluorine was used instead of OF_2 because at temperatures above 175° there is significant decomposition of OF_2 into the elements. This is somewhat of an assumption because the reactions at room temperature and at 125° with an oxygen and fluorine mixture were not tried, and, therefore, it is not known as a fact that the incipient reaction is not that between OF_2 , per se, and CsClF_4 (or ClF_3). At 125° and at room temperature (pressure varying from 1,500 to 10,000 psi) OF_2 did not react with CsClF_4 . At temperatures between 175° and 300° and pressures of 5,000 and 10,000 psi reactions did occur. The major constituents of the reaction products were oxygen and fluorine. In several cases the next most abundant product was ClF_5 . The yield of ClF_5 in one case was 3.4 g which corresponds to about 23% yield based on the fluorine consumed and that which is available from the decomposition of OF_2 . Other volatile products in these reactions were in decreasing order of abundance FClO_2 , FClO_3 , CF_4 , and CF_3OF (the last two probably arising from reaction with Teflon in system). In two cases, in which the Hastelloy-C reactor was used, there were peaks in the infrared spectra attributable to MoF_6 --molybdenum being present in the alloy to an extent of about 16%. The other constituents of the alloy present in a significant amount do not form (at least not very readily) volatile fluorides. The attack on the reactor was quite extensive when Hastelloy-C was the material. The solid products of several experiments were analyzed specifically for corrosion products. These results are summarized in Table VI. Monel ordinarily appeared to suffer very little corrosion and, certainly, would be the material of choice for future high pressure vessels for this type of work.

2. The reaction of oxygen difluoride with tetrafluorohydrazine was accompanied by experimental difficulty. The problem here was that the starting materials reacted either as the reactor was being charged (at -196°) or as the reactor was being warmed from -196° . This was an extremely exothermic reaction. Because the materials had already reacted by the time room temperature was attained, it was decided not to subject the reactor contents to high pressure at elevated temperatures. The products of this reaction were found to be FNO , FNO_2 , NF_3 , OF_2 and what is believed to be a trace of F_3NO .

3. No reaction was observed when CsF was allowed to interact with NF_3 , fluorine with NF_3 , or CsF and fluorine with NF_3 . The conditions were varied such that the pressure ranged from 5,000 to 10,000 psi at temperatures from 160° to 300° .

II. RESULTS AND DISCUSSION

A. The Reaction: Oxygen difluoride with cesium tetrafluorochlorite.

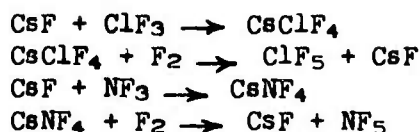
No reaction took place between oxygen difluoride and cesium tetrafluorochlorite until a temperature sufficient to cause decomposition of OF_2 was reached. At this temperature, i.e. about 170° and above, reaction took place giving a mixture of products--those which would be expected if the elements were the starting materials instead of OF_2 . The major products were oxygen and fluorine; however, ClF_5 was produced in good yield (23%) based on the fluorine consumed as compared to that available from the decomposition of OF_2 . Other products isolated and/or identified by infrared spectroscopy were FClO_2 , FClO_3 , OF_2 , CF_4 and an unknown substance which appeared in fractions between -110 and -160 and between -160 and -196 and having absorptions in the infrared at about 7.9μ and 8.17μ . It is believed by this investigator that these are the very strong peaks in trace amounts of trifluoromethyl hypofluorite, CF_3OF (2). Similar absorptions in the infrared spectra have been attributed to a new compound, FClO , (compound "C") reported by the workers at Rocketdyne (3). One may account for the formation of CF_4 and CF_3OF in this work by the attack on the "Teflon" seal tape used on the threads of various connectors and fittings in the system. There was no evidence of the formation of ClF_5O or ClF_3O . The results of these experiments are summarized in Table I.

B. The Reaction: Oxygen difluoride with chlorine trifluoride.

The products of the reaction of OF_2 with ClF_3 are quite like those of OF_2 with CsClF_4 . In this case, however, the yields of ClF_5 were much lower. Furthermore, the more reactive nature of ClF_3 complicated the results because of the extensive attack of the reactors, especially those of Hastelloy-C. Because of the quantity of molybdenum in Hastelloy-C, the possibility exists that the volatile fluoride, MoF_6 may be formed as a corrosion product. This appears to be the case as there is evidence of MoF_6 in the infrared spectra of the room temperature fraction. Conditions and results are also in Table I.

C. The Reactions: Cesium fluoride with nitrogen trifluoride; fluorine with nitrogen trifluoride; and cesium fluoride and fluorine with nitrogen trifluoride.

This series of reactions was investigated because it was thought that one might extend, through the effects of high pressure and temperature, the analogy of the reaction that yields ClF_5 to nitrogen and the formation of NF_5 .



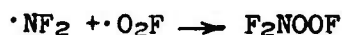
In each experiment made there was at no time any evidence of reaction or complex formation. The starting materials were recovered unchanged. The conditions and results of these experiments are summarized in Tables II, III and IV.

D. The Reaction: Oxygen difluoride with tetrafluorohydrazine.

This reaction was studied with the thought that the hypofluorite bond might be introduced into the molecule.



Another possibility exists here in that $\text{O}_2\text{F}\cdot$ forms at elevated temperatures and pressures and could react with the $\text{NF}_2\cdot$ radical.



Oxygen difluoride does indeed react with N_2F_4 --and, at a temperature not too much above that of liquid nitrogen, an extremely exothermic reaction takes place. The products formed from this reaction were found to be FNO , FNO_2 , NF_3 and a trace of that which appears to have an infrared spectrum characteristic of trifluoramine oxide, F_3NO . Table V summarizes the conditions of this reaction.

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TABLE I. CONDITIONS & RESULTS OF THE REACTION OF OXYGEN DIFLUORIDE WITH CESIUM TETRAFLUOROCHLORITE

<u>CONDITIONS</u>					<u>RESULTS</u>	
<u>T</u>	<u>P, atm.</u>	<u>V, ml.</u>	<u>Time, da.</u>	<u>OF₂(moles)</u>	<u>CsClF₄(moles)</u>	
RT	76	30.0	14	0.083	0.127	Reactants recovered unchanged.
RT	194	30.0	14	0.237	0.135	Reactants recovered unchanged.
RT	337	30.4	12	0.420	0.03	No reaction.
RT	680	11.3	10	0.312	0.03	No reaction.
200°	352	31.0	14	0.272	0.03	-196° fraction v.p. 265 mm Hg., M.W. = 35.7. IR showed only a trace of OF ₂ .
175°	680	11.3	2	0.198	0.03	-110° and above fraction IR showed trace of OF ₂ and FClO ₂ in ClF ₅ . Gas phase fractionated. -196° fraction was predominant. IR showed only trace of OF ₂ -- mostly O ₂ & F ₂ (1:2). -160° fraction OF ₂ with trace-peak due to FClO ₂ and probably ClF ₃ . -110°, -80° fractions essential- ly the same except for trace of FClO ₃ in -110°. ClF ₅ with traces. Unidentified peak at 10.6 μ . -80° fraction is ClF ₅ with traces of FClO ₂ and FClO ₃ .

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TABLE I. CONDITIONS & RESULTS OF THE REACTION OF OXYGEN DIFLUORIDE WITH CESIUM TETRAFLUOROCHLORITE (cont.)

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<u>CONDITIONS</u>					<u>RESULTS</u>
<u>T</u>	<u>P, atm.</u>	<u>V, ml.</u>	<u>Time, da.</u>	<u>OF₂(moles)</u>	
				<u>CsClF₄(moles)</u>	
300°	340	30.0	10	0.217	0.03
<p>Solid phase--purple solid, very low oxidizing power, high nickel content, mildly effervesces in water to give odor like ozone and green ppt. (NiF₂·4H₂O) with resulting yellow-green soln. F-content 31%.</p> <p>The volatile products fractionated in much the same way as did the experiment above (175° and 680 atm.). There was, however, more CF₄ present probably due to Teflon seal tape on the valve threads.</p> <p>The solid products were also quite similar in behavior except that a purple solution resulted when placed in water. F⁻ content was about 31%. The main difference must lie in attack of the Monel (as opposed to Hastelloy-C above).</p> <p>Volatile products are O₂, F₂, CF₄, FClO₂, SiF₄, and ClF₃. Unidentified peaks present in the infrared spectra, probably due to MoF₆.</p> <p>Volatile products are O₂, F₂, OF₂, CF₄, ClF₃, CF₃OF, FClO₂, FClO₃ and ClF₅.</p>					
300°	680	11.3	6	0.164	0.03
175°	340	30.0	12	0.105	0.105

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TABLE II. CONDITIONS & RESULTS OF THE REACTION OF NITROGEN TRIFLUORIDE WITH CESIUM FLUORIDE

<u>CONDITIONS</u>				<u>RESULTS</u>		
<u>T</u>	<u>P, atm.</u>	<u>V, ml.</u>	<u>Time, d.</u>	<u>NF₃(moles)</u>	<u>CsF(moles)</u>	
160°	340	30.0	4	0.287	0.046	No reaction. NF ₃ only in IR.
300°	340	30.0	4	0.220	0.046	No reaction. NF ₃ , trace NO ₂ in IR.
160°	680	11.3	3	0.198	0.046	No reaction. NF ₃ only in IR.
300°	680	11.3	4	0.163	0.046	No reaction.

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TABLE III. CONDITIONS & RESULTS OF THE REACTION OF NITROGEN TRIFLUORIDE WITH FLUORINE

				<u>NF₃(moles)</u>	<u>F₂(moles)</u>	
160°	340	30.0	5.5	0.144	0.43	No reaction.
300°	340	30.0	5.5	0.109	0.108	No reaction.
160°	680	21.75	5.5	0.208	0.208	No reaction.
300°	680	21.75	3.5	0.158	0.158	No reaction. Trace MoF ₆ ? in IR.

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TABLE IV. CONDITIONS & RESULTS OF THE REACTION OF NITROGEN TRIFLUORIDE, FLUORINE AND CESIUM FLUORIDE

T	<u>CONDITIONS</u>					<u>RESULTS</u>
	<u>P, atm.</u>	<u>V, ml.</u>	<u>Time, da.</u>	<u>NF₃(moles)</u>	<u>F₂(moles)</u>	<u>CsF(moles)</u>
300°	340	30.0	3.5	0.11	0.11	0.046
300°	680	19.75	3	0.15	0.15	0.046
						No reaction.
						No reaction.

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TABLE V. CONDITIONS & RESULTS OF THE REACTION OF OXYGEN DIFLUORIDE WITH TETRAFLUOROHYDRAZINE

<u>V, ml.</u>	<u>OF₂(moles)</u>	<u>N₂F₄(moles)</u>	Exothermic reaction takes place just after removal from LN ₂ bath. Products found were FNO, FNO ₂ , OF ₂ and a trace of what is believed to be F ₃ NO.
13.3	0.067	0.067	

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TABLE VI. ANALYTICAL RESULTS OF SOLID PRODUCTS IN %

Sample # Element	I	II	III	IV	V	VI	VII*	VIII*
F	31.5	29.7	36.8	44.0	32.6			
Cs	34.9	43.8	23.2	0.0	36.8			
Ni	20.7	16.7	28.4	41.5	13.7			
Cu	10.56	0.09	0.06	0.01	0.01	86.56		54-55
Cr	nil	2.98	4.62	4.22	5.93			--
Mo	0.21?	0.14	0.13	0.09	0.36		63.0-70.0	14.5-16.5
Fe	0.35	1.56	2.51	3.27	1.99		24.7-31.7	15-17
W	--	0.26	0.14	0.19	0.18		2.50 max	4-7
V	0.	0.	0.	0.1	0.24			3-4.5
Mn	0.21	0.14	0.24	0.36	0.19			0.35 max
Co	0.71	0.22	0.38	0.52	0.24		2.00 max	1 max
Total	97.14	95.59	96.48	94.26	92.24		included as Ni	2.5 max
							100.00	

I	OF2 + CsClF4	-- in Monel
II	OF2 + CsClF4	-- in Hastelloy-C
III	OF2 + CsClF4	-- "
IV	OF2 + ClF3	-- "
V	OF2 + CsClF4	-- "
VI	CsF	-- starting material
VII	Monel 400	-- typical analysis taken from literature
VIII	Hastelloy-C	-- typical analysis taken from literature

*The trace constituents--Si, C, and S--are not given in this table as they were not determined. These elements in toto account for less than 1%.

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III. EXPERIMENTAL PROCEDURESA. General:

The experimental procedure used was the same for each reaction studied. The volume of each reactor was determined and then charged with the least volatile reactant. In cases where one of the starting materials was a solid, the resulting decrease in free volume was determined by using the density of the solid to determine its volume and subtracting that amount from the volume of the reactor. The quantity of gas required to generate the pressure desired at a given temperature was calculated using the corrected volume in the ideal gas law. In some cases the reactor was a Hoke 4HSM-30 cylinder fitted with a Hoke 10115-2 valve. Both valve and cylinder are rated for at least 5,000 psia and are of Monel. In the experiments in which the pressure exceeds 5,000 psi, one of two reactors of Hastelloy-C was used. These two reactors were of the same type construction except that one was made by Pressure Products Industries, Hatboro, Pa. and the other was made by Autoclave Engineers, Erie, Pa. One had a volume of 13.3 ml. whereas the other was 21.75 ml. The reactor was a high-pressure valve with a high-temperature packing extension. The high-pressure side of the valve had a length of tubing of dimensions to give the rating desired and suitable volume connected to it. The tubing then was capped off with a high-pressure blind-gland to form the autoclave. Both bodies (i.e., the tubing) were machined from round stock with the length being varied and a slight change in ID to give the different volumes. A graphical description of a similar reactor is given in the annual report (1).

A typical experiment is described as follows:

The reactor was charged with CsClF_4 and assembled. It was then connected to a Monel-nickel vacuum manifold (1), evacuated, and weighed. After this, the reactor was reconnected to the manifold and condensed into the reactor using liquid nitrogen (LN_2). By using the gas law and the calibrated volume of the manifold, a very close estimate could be made of the amount of OF_2 being added to the reactor. The reactor then was reweighed to determine the weight of OF_2 added. The reactor was then placed in an oven or furnace set at the desired temperature for several days. After the reaction period, the contents of the cylinder were separated by usual low-temperature fractionation techniques. The various fractions were analyzed by infrared spectroscopy. In some cases molecular weights were determined.

B. Analytical Methods:

1. Fluorine: After appropriate preparation, the fluoride was distilled from sulphuric acid. The distillate was buffered to a pH of 3.2 and titrated with standard $\text{Th}(\text{NO}_3)_4$ to alizarin Red-S end point.

2. Metals: These were determined using a Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer.

3. Infrared Spectra: Spectra were made using a Perkin-Elmer Model 337 Infrared Spectrophotometer, 2.5 to 25 μ . The gas cell was of nickel and had a path of 5 cm. The windows were AgCl and were fitted to the cell with Kel-F Wax.

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IV. REFERENCES

1. Annual Technical Summary Report, Synthesis of New Fluorine Compounds (U), Ozark-Mahoning Co., Tulsa, Oklahoma, March 1966, DDC Nr. Ad-480 450. UNCLASSIFIED.
2. Lagemann, R. T., Jones, E. A. & Woltz, P. J. H., J. Chem. Phys. 20, 1768 (1952).
3. R-6641, Annual Report, Inorganic Halogen Oxidizers, Rocketdyne, A Division of North American Aviation, Inc., Canoga Park, California, 31 July 66, CONFIDENTIAL.

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13. ABSTRACT Reactions were studied in which high pressure and elevated temperatures were employed. Pressures ranged from 340 to about 680 atm. The reactions studied were as follows: OF ₂ with CsClF ₄ , OF ₂ with ClF ₃ , OF ₂ with N ₂ F ₄ , CsF with NF ₃ , fluorine with NF ₃ , and CsF and fluorine with NF ₃ .	

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